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APPLICATION NUMBER: *60/459,867*
FILING DATE: *April 01, 2003*
RELATED PCT APPLICATION NUMBER: *PCT/US03/21267*

By Authority of the
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04-03-03 60459867 .040103

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JC904 U.S. PTO 04/01/03

RJP:cms .04/01/03 . . 180174.doc
Attorney's Matter No. 3468-64343/RJP
PATENT

EXPRESS MAIL LABEL NO. EV 295231926 US
DATE OF DEPOSIT: April 1, 2003

11002 U.S. PTO 60/459867 04/01/03

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BOX PROVISIONAL PATENT APPLICATION
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PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 C.F.R. § 1.53(c).

TITLE: PROCESS FOR THE TREATMENT OF WASTE METAL CHLORIDES

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Last	First	MI	City, State or Foreign Country and City

☒ 12 pages of specification and 3 pages of claims enclosed.

☒ 1 sheet(s) of drawings are enclosed.

☒ Provisional Filing Fee Amount:
- ☒ $160, large entity
- ☐ $ 80, small entity

☒ A check in the amount of $160.00 to cover the filing fee is enclosed.

☒ The Director is hereby authorized to charge any additional fees which may be required in connection with the filing of this provisional application and recording any assignment filed herewith, or credit over-payment, to Account No. 02-4550. A copy of this sheet is enclosed.

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PROCESS FOR THE TREATMENT OF WASTE METAL CHLORIDES

The present invention relates to a method for reclaiming valuable and useful materials from the residues of silicon hydrochlorination, chlorosilane distillation, and other processes.

Chlorosilanes such as trichlorosilane and silicon tetrachloride are prepared by reacting crude silicon with chlorine or hydrogen chloride. Trichlorosilane can also be prepared by the reaction of silicon tetrachloride and hydrogen with crude silicon. In common industrial processing the crude silicon is of the type which has a silicon content greater than about 85% by weight[1,2]. The impurities in the crude silicon are mainly iron, aluminum, calcium and titanium which are converted to their respective chlorides in an analogous method as the production of the chlorosilanes. In addition to these metals, other purposefully added metals may be present as catalysts and promoters. Such added active metals are copper, zinc, silver and nickel. Finally, during the distillation purification of the chlorosilanes a residue fraction is generated. This residue can contain fine particles of silica, higher boiling polychlorosilanes and traces of high boiling organic materials that may be used as catalysts or promoters in other parts of the chlorosilane production process.

Customarily, the residues that result from distillation conditions are presented in the form of a slurry or suspension of solids and higher boiling liquids containing sufficient chlorosilanes to maintain fluidity. This stream requires additional processing to render it non-reactive or non-hazardous before it can be ready for environmentally safe disposal.

The distillation of the chlorosilanes is carried out as completely as possible because any chlorosilanes remaining in the residue can no longer be converted into useful products

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[1] US Pat. 3,878,291 Keller
[2] US Pat. 4,676,967 Breneman

and therefore represent a loss in value. In those instances where the residues to be disposed of are in the form of a suspension, the solid fraction consists of unreacted silicon metal, silica and other metals and metal chlorides. The solids are slurried in a liquid phase which contains 50 – 80% silicon tetrachloride and/or trichlorosilane and 1 – 30% hydrochloropolysilanes. This stream may be further concentrated in a screw-conveyor or paddle type drier to recover essentially all of the silicon tetrachloride and trichlorosilane, leaving a solid powder residue containing the metal chlorides, unreacted silicon metal and the traces of silica, non-volatile organics and the like[3].

Various means have been disclosed to render the solid residue suitable for environmentally safe disposal. German Patent 21 61 641 discloses the reaction of a chlorosilane distillation residue with water vapor accompanied by the formation of hydrogen chloride. However, an adequate reaction takes place only with a stoichiometric excess of water vapor so that additional hydrochloric acid is produced from the unreacted water which then has to be disposed of.

To avoid the formation of additional hydrochloric acid, German Patent 36 42 285 proposed to perform the hydrolysis in the presence of additional hydrogen chloride and recycle the unreacted water.

U.S. Patent 4,690,810 discloses a process for the reaction of the chlorosilane residues with milk of lime to form a slurry of soluble calcium chloride and solid metal hydroxides and oxides. That process does not allow for reclaiming any of the valuable chlorosilanes required to provide fluidity to the residue and further requires a means to convert the calcium chloride solution into a commercial form, else adding to the already great environmental load.

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[3] US Pat. 4,892,694 Rtizer

Other means have been proposed to treat residues from the purification of chlorosilanes such as are generated during the production of polycrystalline silicon. Those processes involve hydrolysis of the residues, but neutralization of the resulting hydrochloric acid and filtration to remove the co-product silica both involve the use of expensive acid resistant equipment and the high maintenance costs associated with the processing of corrosive hydrochloric acid. Filtration of the resulting slurries is difficult and many times is just not possible as the hydrolysis reactions form gels and ultra-fine particles. All of these present processes, without exception, result in contacting the residue with liquid water. The reaction of water with either the residual chlorosilanes or the metal chloride impurities results in the formation of corrosive hydrochloric acid. Therefore, the process equipment must be constructed of corrosion resistant materials. Leaks and spills provide a high likelihood of environmental contamination and worker exposure to corrosive materials. Furthermore, the aqueous hydrolysis of chlorosilanes results in the formation of silica solids not only within the reaction mixture, but the solids can deposit on the interior portions of the equipment causing a process limiting build-up or plugging of pipelines.

Summary of the Disclosure

It would be useful to provide a method for economically processing the residues from chlorosilane production to yield a waste product that can be readily disposed of, and preferably, to completely recover valuable chlorosilanes. It also would be desirable for the process to afford an opportunity to reclaim valuable metals by well known extractive metallurgy techniques. It further would be useful to provide a process which can be conducted without need for equipment constructed of the exotic metals or materials required to be resistant to the corrosion of hydrochloric acid.

Described herein are processes by which such residues can be dried and the volatile chlorosilanes (or titanium chlorides) recovered for re-use while the non-volatile solids, containing reactive metal chlorides, are treated with an alkali carbonate or bicarbonate

humectant to produce a non-fuming, substantially neutral or slightly basic solid. That solid is suitable for environmentally safe disposal. Alternatively, the residue may be further processed by extractive metallurgy methods to recover valuable metals.

One particular method described herein proceeds without the formation of a liquid waste product and comprises:

1) Evaporating the volatile chlorosilanes in a suitable continuous or batch type drier, optionally, in the presence of a chloride complexing agent,

2) Condensing the evaporated chlorosilanes and making them available for complete recovery and re-use, thereby significantly increasing the overall yield, and

3) Subjecting the substantially non-volatile solid residues to the action of selected alkaline hydrate solids at a temperature in excess of about 80°C to yield a stable, substantially neutral or somewhat basic solid suitable for disposal or precious metal recovery.

The naturally occurring mineral trona is a usable alkaline hydrate material. Trona is inexpensive, readily available, and environmentally benign. A particularly useful, mechanically refined trona is sold under the trademark "T-200®" by Solvay Mineral, Green River, WY. It is identified by CAS number 6106-20-3. Its chemical composition is nominally sodium carbonate (CAS 0497-19-8) 46%, sodium bicarbonate (CAS 0144-55-8) 36% and water (CAS 7732-18-5) 16%. T-200 trona is a powder having the following size characteristics:

Sieve Opening	Typical Weight Percent
<70 µm	75
<28 µm	50
<6 µm	10

Brief Description of Drawing

The drawing figure is a schematic flow sheet of a process for the treatment of waste metal chlorides.

Detailed Description

A stream of flowable solids-laden chlorosilane to be treated (1) originates from the hydrogenation of silicon tetrachloride over a fluidized bed of metallurgical silicon, or from the hydrochlorination of silicon metal in a fluidized bed reactor using hydrogen chloride, or from the residues of the distillation processes that purify trichlorosilane and silicon tetrachloride produced from these reactions.

One or more of these streams can be combined into an agitated slurry collection vessel (3) that serves as an intermediate storage vessel prior to feeding the slurry (5) to the treatment system. The composition of the slurry can vary considerably, but may consist of components as listed in Table 1.

Table I

Typical composition of waste chlorosilane/solid residue slurry

Liquid Fraction, wt%	77.6
Trichlorosilane	0.2
Silicon tetrachloride	83.6
Cl_6Si_2O	14.2
Solid Fraction, wt%	22.4
Silicon (elemental)	54.6
Silica	19.1
Chloride	16.1

In the illustrated method, the crude slurry is flowed (5) into a batch drier vessel (7) equipped with paddle type mixer, bag filter (8), heating jacket, and solid discharge valve (12). Other mechanical methods of conducting the evaporation of the volatile chlorosilanes are possible and this example is not meant to limit the scope of the invention.

The evaporation/concentration can be enhanced if a complexing agent is added to reduce the volatility of the aluminum chloride component in the solid residue mixture. A

readily available and well known complexing agent is finely ground sodium chloride[4]. The finely milled sodium chloride can be added to the charge of slurry. The amount of sodium chloride added is nominally at least twice the weight of the estimated amount of aluminum chloride contained in the remainder of the slurry. The sodium chloride is useful in forming a chemical complex with the aluminum chloride contained in the slurry. The salt complex lowers the vapor pressure of the aluminum chloride and thus retains the aluminum chloride with the slurry solids as the volatile chlorosilane fraction is evaporated.

The charge of volatile chlorosilanes and mixed solids is sufficiently heated by a heating medium in the jacket of the evaporator to gasify at least a portion of the chlorosilanes; and the volatile chlorosilanes are removed (14) as a vapor. The chlorosilanes vapors (16) are condensed (9) and collected in a recovery vessel (10). A bag filter (8) may be employed on the evaporator to reduce the carry-over of fine particles with the chlorosilane vapors. In a preferred mode of operation, the evaporator may be recharged several times after the bulk of the chlorosilanes have been evaporated until the accumulated solids amount to about ¼ of the working volume of the evaporator. At this point, the temperature of the drier is raised to complete the evaporation of the chlorosilanes, which, at atmospheric pressure, is a temperature of about 70°- 80°C.

The chlorosilanes collected in the receiver (10) may then be returned (13) to the refining section of the chlorosilane production unit.

The vent from the evaporator (14) is then switched to allow the vent gases (15) to pass to a suitable water spray scrubber (11) or similar treatment unit that is designed to remove residual amounts of hydrogen chloride from the vent gas stream.

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4 Fannin, A. A.; King, L. A.; Seegmiller, D. W.; Oye, H. A. *J. Chem. Eng. Data* 1982, *27*(2), 114-119.

A charge of finely milled trona, natural sodium sesquicarbonate (2) is added to the evaporator (7) from a storage bin (4) via a lock chamber (6). The amount of trona to be added is such as to provide a pH greater than 7 in the residue solid. The optimum amount of trona to be added is generally determined by experiment since the composition of the residue material can vary. A modest excess of trona is desirable, but a greater excess presents only a minor additional cost. The mixture of dry solids and trona is heated to a temperature of between about 120° and 150°C, although higher temperatures may be used without negative effect. During the heating, hydrated moisture in the trona reacts with the metal chlorides and traces of chlorosilanes. Some HCl is formed, which reacts with the sodium carbonate portion of the trona. Additionally, as the trona is heated, it thermally decomposes to release additional moisture and carbon dioxide gas. The decomposition of the trona results in a porous solid which can readily react with the released hydrogen chloride gas. The released gas, mainly carbon dioxide, un-neutralized hydrogen chloride and excess moisture is vented to the scrubber (11).

The neutralized flowable solid material consisting of the excess and decomposed trona, silicon metal, silica, and neutralized or hydrated metal chlorides are then cooled to a safe handling temperature and discharged (12). Provided sufficient trona has been used, the pH of a 10% slurry of the solid is between 7 and 10.5 and no odor of hydrogen chloride is present.

The neutralized flowable solids may be disposed of in a suitable landfill, or made available for recovery of selected metals using conventional extractive metallurgy methods.

Examples of suitable alkaline hydrates that may be used in the process are sodium or potassium sesquicarbonate, sodium aluminum sulfate dodecahydrate, sodium acetate trihydrate, sodium ammonium phosphate tetrahydrate, sodium carbonate decahydrate, sodium citrate dehydrate, sodium dihydrogen phosphate dehydrate, and mixtures of calcium carbonate or sodium carbonate, sodium bicarbonate, and/or other basic salts. In addition,

inert hydrated minerals may be used such as Aluminite, Apophyllite, Bloedite, Chabazite, Gaylussite, Gmelinite, Heulandite, Kainite, Kieserite, Laumonitite, Levyne, Mesolite, Mirabilite, Montmorillonite, Mordenite, Natrolite, Newberyite, Phillipsite, Scolecite, Stilbite, Struvite, and damp soil. In the case of damp soil, excess water content can cause processing difficulties; a water content of about 5% (w/w) is suitable for most purposes. Soil may or may not be alkaline depending on the where it is acquired. In order to satisfy the requirements for non-hazardous land fill disposal, the basic anion(s) is (are) generally limited to sodium, potassium, calcium, and magnesium and excludes lithium, rubidium, barium, strontium, and the like. These materials are milled or otherwise processed, if necessary, so that they are provided in a flowable solid form, typically as a powder or granules.

Although not to be bound by theory of operation, it is believed that the successful working of the disclosed processes depends on the water trapped in the solid hydrate. The trapped water is not released until it is exposed to the "waste" which contains, e.g., aluminum chloride and iron chloride, and traces of residual chlorosilanes. Upon exposure to metal chlorides in the waste, the hydrate is at least partially dehydrated by a transfer of water to the metal chlorides. The transferred water forms aluminum chloride hydrate (for example) and silica. The amount of the hydrate supplied and the water content thereof should be chosen to be sufficient to completely hydrate all the metal chloride in the waste.

As there is a small amount of HCl also liberated during this reaction and subsequently during long term exposure, it is best to react the HCl with an alkaline salt to neutralize at least a portion of the hydrogen chloride and thereby increase the pH of the mixture. The alkaline salt may be provided by using an alkaline hydrated mineral to react with the metal chlorides, or a separate alkaline salt may be provided. For example, in the alkaline hydrated mineral trona, sodium carbonate and bicarbonate are present in sufficient excess to serve as alkaline salts that react with the hydrogen chloride and form harmless salt, water and carbon dioxide. Some naturally occurring hydrated materials, such as "soil" for

example, may or may not be alkaline depending on the where they are acquired, and thus might or might not benefit from the use of a separate alkaline salt. Calcium carbonate and magnesium hydroxide are examples of separate alkaline salts that could be added to neutralize HCl. Such materials are best introduced as flowable solids, such as powder or granules. If an excess of a salt is provided, the resulting mixture will be somewhat basic.

The resulting neutralized residue also is in the form of a flowable solid material, typically powder or granules, and can be safely disposed of in an environmentally acceptable manner. After discharge of the neutralized solids, the mixer/drier is ready for a subsequent charge of chlorosilane slurry with out need for further clean-up. .

The discharged solids meet the requirements for non-hazardous solid waste by the "TCLP" or Toxic Characteristic Leaching Protocol[5]. Alternatively, if valuable metals, such as copper, nickel or silver and the like, are used as catalysts or promoters in the production of chlorosilanes, the neutralized flowable residue solids can be made available for recovery of those metals by conventional hydrometallurgy extraction techniques.

Because the moisture is carried into the process in the form of a hydrated solid, there is substantially no free moisture within the process. The metal chlorides, for example aluminum chloride, have a much stronger affinity for moisture than the alkali carbonate hydrate. Thus the environment within the mixer/drier is maintained in a state where no condensation of water or hydrochloric acid occurs, thus reducing the corrosive effect. The mixer/drier can be constructed of a duplex stainless steel alloy such as Ferillium that is much less expensive than the nickel/chromium/molybdenum alloys or glass enameled equipment that would otherwise be required.

Alternatively, the solid may be sold to a hydrometallurgy extraction processor where the valuable metals could be selectively reclaimed.

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[5] 40 CFR 268.49

For example, if the alkali carbonate hydrate used in the process were trona, natural sodium sesquicarbonate, washing the solid with water would remove the bulk of the sodium carbonate and sodium chloride. Then the remaining solid could be acidified with sulfuric acid to form soluble copper sulfate. The copper sulfate could then be extracted by an organic solution of an oxime in kerosene as described in U.S. Pat. 6,242,625.

Example 1

1,160 Kg of a slurry consisting of 25% solid silicon and metal chlorides and 75% of a mixture of silicon tetrachloride and trichlorosilane was added to a horizontal mixer/drier constructed of Ferillium duplex stainless steel and having a processing volume of 3.24 m^3. 36 Kg of Cargill Microsized 66 micronized sodium chloride was also added. At essentially atmospheric pressure, heat was applied to the jacket of the mixer/drier and the bulk of the chlorosilanes was boiled off and condensed into a receiver. When the batch temperature began to rise above 60°C (the boiling point of silicon tetrachloride at process pressure, a fresh charge of 1,160 kg of slurry was made and the boiling continued. This fill, boil, fill sequence was repeated until a total of 4,211 kg of slurry had been charged. After the last slurry charge, the mixer/drier temperature was allowed to rise to 80°C to complete the evaporation of the chlorosilane. The mixer/drier vent was switched to a water spray vent scrubber and a charge of 250 kg of Solvay® T-200 finely ground trona, natural sodium sesquicarbonate, was added to the mixer/drier. The temperature of the mixer/drier was raised to 130°C over a period of one hour and held there for an additional two hours to assure complete reaction. The batch was cooled to less than 50°C and a fine gray powder solid was discharged to a bin. A 10% slurry of the powder in water indicated a pH of 10.3. The powder did not present any acidic odor, was free flowing and did not ignite when heated in air.

Example 2

A slurry consisting of 110 gram of solid residue from the hydrochlorination of silicon and 200 ml of silicon tetrachloride was placed in a 500 ml agitated flask that was fitted with several small TFE discs in the vapor path before a condenser. The slurry was gently heated to 80°C while the silicon tetrachloride was evaporated. 18 gram of sodium sesquicarbonate powder was added to the flask and the temperature was increased to 130°C. After holding the temperature for two hours, the flask was cooled and the residual waste product had an indicated pH of 10.4. During the heating cycle, a yellow/white fume was collected on TFE discs in the cooler portions of the apparatus. 160 mg of fume consisting of > 90% aluminum chloride with a minor amount of iron chloride were collected on the TFE discs.

Example 3

A slurry consisting of 110 gram of solid residue from the hydrochlorination of silicon (containing 5.4% Al, 2.6% Fe), 15 gram of finely ground sodium chloride and 200 ml of silicon tetrachloride was placed in a 500 ml agitated flask fitted with several small discs of TFE mounted in the vapor path below the condenser. The flask was heated slowly to evaporate the silicon tetrachloride. When the temperature reached 63°C, no more vapors were being removed. Then 30 g of Solvay T-200 finely ground trona (natural sodium sesquicarbonate) were added and the heating continued up to 160°C. After cooling, the residual solids were free flowing and odor free. The pH was 9.9. During the heating cycle, there was a markedly lower amount of white fume noticed. The amount of fume collected on the TFE discs was reduced to 8.5 mg of aluminum chloride (from 160 mg in Example 2).

While the foregoing description and examples relate primarily to the treatment of residues of silicon hydrochlorination and chlorosilane distillation processes, it should be

appreciated that the methods described herein have broader applicability. The methods are also suitable for treatment of residues from other metal chlorination processes, such as the chlorination of rutile to produce titanium chlorides and the chlorination of silica to produce silicon tetrachloride. Those processes result in the co-production of iron and aluminum chloride which are generally regarded as waste materials and must be neutralized prior to disposal. The residues are dried and the volatile chlorosilanes (or titanium chlorides) are recovered for re-use while the non-volatile solids, containing reactive metal chlorides are treated with an alkali carbonate or bicarbonate humectant to produce a non-fuming, substantially neutral or somewhat basic solid. The neutralized solid material is suitable for environmentally safe disposal. Alternatively, the residue may be further processed by extractive metallurgy methods to recover valuable metals.

Claims:

1. A method of processing the residue from a chlorosilane manufacturing process, the method comprising:

concentrating a residue mixture containing volatile chlorosilanes and lower volatility components including at least one metal chloride in a drier unit suitable for processing a solid fraction;

separating volatile chlorosilane vapors from the mixture for recycle or reprocessing;

contacting the remaining substantially chlorosilane-free solid residue with a hydrate at a temperature from 80° to 150°C such that the at least one metal chloride reacts with the hydrate; and

discharging the resulting flowable mixture for disposal or metals recovery.

2. The method of claim 1 further comprising contacting the substantially chlorosilane-free solid residue with an alkaline salt to increase the pH of the resulting flowable mixture.

3. The method of claim 2 further comprising simultaneously contacting the remaining substantially chlorosilane-free solid residue with a hydrate and contacting the remaining substantially chlorosilane-free solid residue with the alkaline salt.

4. The method of claim 3 wherein the contacting of the remaining substantially chlorosilane-free solid residue with a hydrate and the contacting of the remaining substantially chlorosilane-free solid residue with the alkaline salt is accomplished by contacting of the remaining substantially chlorosilane-free solid residue with mechanically refined trona, which is a natural form of sodium sesquicarbonate, is a hydrated mineral, and provides an alkaline salt.

5. The method of claim 2 wherein:
the alkaline salt is calcium carbonate; and
the hydrated mineral is damp natural soil.

6. The method of claim 2 wherein:
the alkaline salt is magnesium hydroxide; and
the hydrated mineral is montmorillonite clay.

7. A method of processing the residue from a chlorosilane manufacturing process, the method comprising:

concentrating a residue mixture containing volatile chlorosilanes and lower volatility components including at least one metal chloride in a drier unit suitable for processing a solid fraction in the presence of finely milled sodium chloride;

separating volatile chlorosilane vapors from the mixture for recycle or reprocessing;

contacting the remaining substantially chlorosilane-free solid residue with a hydrate at a temperature from 80° to 150°C such that the at least one metal chloride reacts with the hydrate; and

discharging the resulting flowable mixture for disposal or metals recovery.

8. The method of claim 7 further comprising contacting the substantially chlorosilane-free solid residue with an alkaline salt to increase the pH of the resulting flowable mixture.

9. The method of claim 8 further comprising simultaneously contacting the remaining substantially chlorosilane-free solid residue with a hydrate and contacting the remaining substantially chlorosilane-free solid residue with the alkaline salt.

10. The method of claim 9 wherein the contacting of the remaining substantially chlorosilane-free solid residue with a hydrate and the contacting of the remaining substantially chlorosilane-free solid residue with the alkaline salt is accomplished by contacting of the remaining substantially chlorosilane-free solid residue with mechanically refined trona, which is a natural form of sodium sesquicarbonate, is a hydrated mineral, and provides an alkaline salt.

11. The method of claim 8 wherein:
the alkaline salt is calcium carbonate; and
the hydrated mineral is damp natural soil.

12. The method of claim 8 wherein:
the alkaline salt is magnesium hydroxide; and
the hydrated mineral is montmorillonite clay.

13. A method of processing a flowable solid material that includes at least one metal chloride, the method comprising:

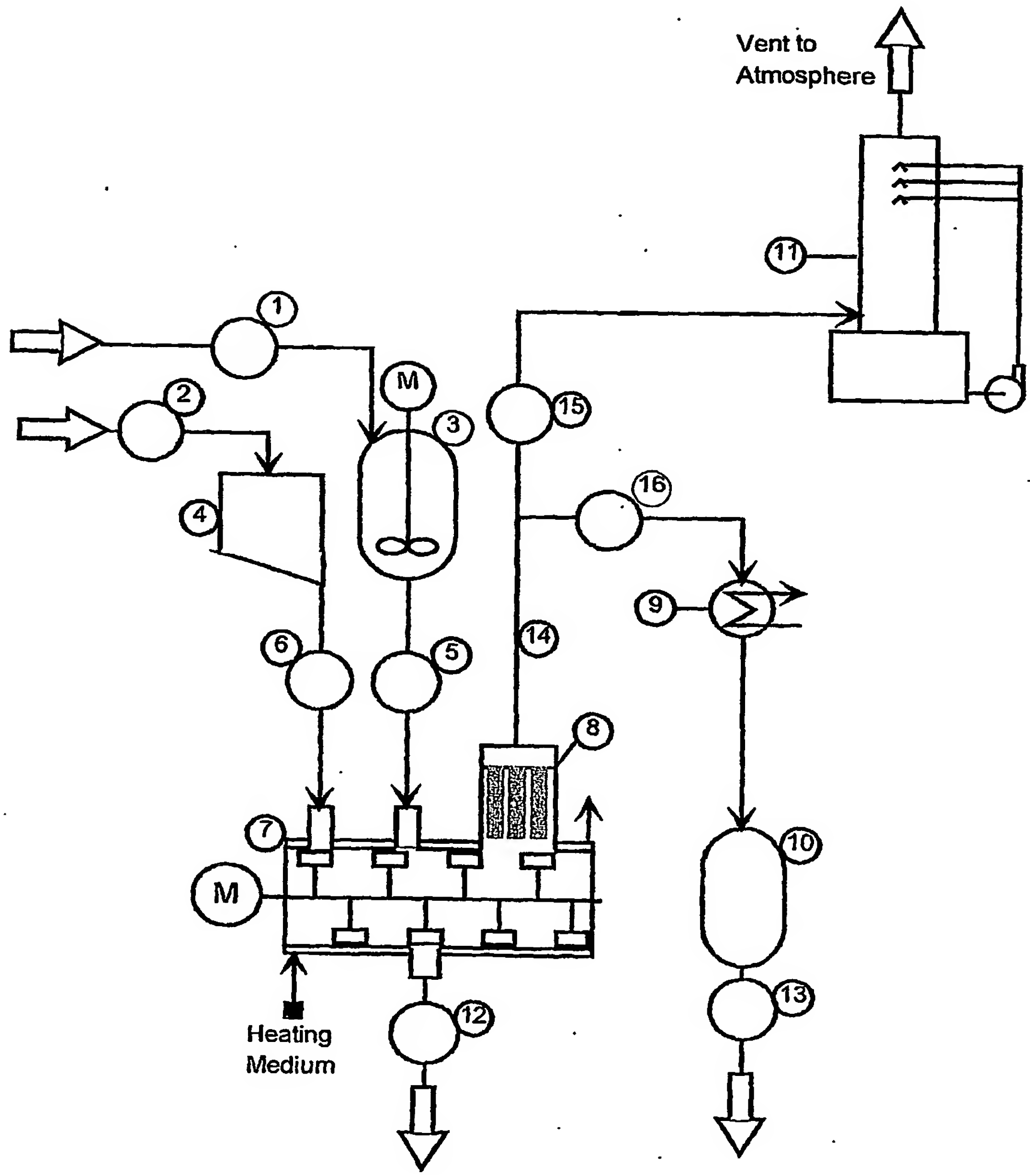
contacting a flowable solid material that includes at least one metal chloride with a flowable solid hydrate in a vessel at a temperature from 80° to 150°C such that the at least one metal chloride reacts with the hydrate; and

discharging the resulting flowable mixture of solids for disposal or metals recovery.

PROCESS FOR THE TREATMENT OF WASTE METAL CHLORIDES

Abstract of the Disclosure

A process is described for treating the residues from the hydrochlorination of silicon and from chlorosilane distillations, wherein the volatile chlorosilane components are recovered for re-use and the non-volatile residue is reacted with a neutralizing humectant. The resulting solid material is suitable for land-fill disposal or recovery of valuable metal constituents by extractive metallurgy techniques.


Vent to
Atmosphere
11
1
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12
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Heating
Medium

Figure 1